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HIGH TEMPERATURE ION SOURCE AND THERMOHM DEVELOPMENT FOR STABLE ISOTOPE PRODUCTION

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ABSTRACT

This report describes the development of an ion source unit and thermohm for operation in the range of 650 - 1000°C.

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INTRODUCTION

For several years, the Stable Isotopes Division has received requests from various sources for the isotopes of the rare earths and other elements for which no charge materials were available which would operate in modified Beta ion source units. Due to the lack of suitable ion sources for use in processing these materials, none of these isotopes had been made available.

A survey of the literature indicated that the rare earth halides have sufficient vapor pressure for calutron charge materials in a range from 800°C to 1000°C. This is considerably above any temperature obtainable using standard Beta ion sources; consequently a new source has been designed. This report covers the design and development of a source to operate with charge bottle temperatures from 650°C to 1000°C.

PREVIOUS EXPERIENCE

The first attempt to run a rare earth charge material was made using a source in which the charge material ($CeCl_3$) was heated by electron bombardment. This ion source was unsuccessful due to poor temperature regulation.

A second attempt was made using M14 type source units obtained from the Beta Development Group. These units had two arc chambers and were designed for uranium tetrafluoride (UF_4) charge material with a temperature range extending up to about 900°C. Since only one arc is used in stable isotope opera-

tions these sources required minor modifications for operation with cerium.

Operation with the reworked M14's, although much better than previous rare earth runs, was still unsatisfactory. The major difficulties were:

1. Charge bottle leakage which resulted in heater shorts.
2. Difficult maintenance of the heaters due to fracturing of the molybdenum elements.
3. Thermohm (resistance thermometer) failures at the elevated temperatures.
4. Charge condensation in the vapor manifold and arc chamber.

In an attempt to obtain a more satisfactory source, the M14 unit was redesigned. Separate oven and arc chamber heaters were provided so that the arc region and the vapor manifold temperatures could be kept sufficiently high to prevent charge vapor condensation. The heaters consisted of molybdenum wire cast in alundum cement (RA-162). The length and size of the molybdenum elements were calculated to utilize the full output of the largest power supplies available, i.e., 3600 watts on the oven supply and 1800 watts on the arc chamber heater supply. Since operating temperatures up to 1200° were desired, a special thermohm was required; furthermore, provisions to permit thermocouple temperature regulation if the new thermohm proved unsatisfactory were desirable.

PRELIMINARY HEATER TESTS

Since heaters of the proposed type had not been used previously, a series of tests were made to determine their characteristics. The various

heaters were tested in a vacuum by surrounding them with heat shielding, supplying measured quantities of power to them, and measuring the temperature at the first heat shield with a thermocouple. All of the preliminary tests were made to determine the maximum temperature which could be attained at various power input levels. At the same time, resistances of the heaters at these various temperatures were measured and compared to resistances permitting maximum utilization of available power supplies. None of the heaters failed during these tests, and temperatures in excess of 1500°C were attained as evidenced by the melting of the several layers of stainless steel heat shielding nearest the heaters.

HIGH TEMPERATURE THERMOHM DEVELOPMENT

With temperatures of this order available, it was evident that a special thermohm would be required; thus tests proceeded simultaneously on both heater and thermohm development in order to determine the operating characteristics of the thermohm and to locate it properly with respect to the heater elements and charge bottle.

Since the primary reason for failure of the standard thermohm at elevated temperature is exfoliation of the insulating mica, the first need in the new thermohm was for an insulating material which would be stable at temperatures up to 1200°C. A second requirement was for a case and insulator which would be stable in vacuum at the high temperatures anticipated since poisoning of the platinum resistance element had to be prevented if reproducible results were to be obtained.

Certain physical dimensions were also restricted in order to retain as many of the original design features of the unit as possible.

The insulating material first considered was pure fused Al_2O_3, but fabrication difficulties prevented its use. A magnesium oxide compound, magnerite, was tried and found unsatisfactory. The final thermohm design consisted of a .003" platinum wire wound non-inductively on a MgO spool in a double 3/8-20 thread. The spool was enclosed in a cylindrical case of purified graphite having a wall thickness of 1/32." This assembly was approximately 1/2" in length and was inserted in the back of the graphite oven housing so that the end of the thermohm case was exposed to the charge bottle. A MgO coupling was used between the graphite case and stainless support tube, thereby reducing heat transfer by conduction and causing the thermohm to approach more closely the bottle temperature. Figure 1 shows the thermohm assembly.

This type of resistance thermometer has been used at temperatures of 1000°C without failure and is presently used with a special micromax reworked to regulate oven temperatures of that order. Figure 2 is a performance curve of the high temperature thermohm and indicates that its response is virtually linear over the desired range.

HEATER DEVELOPMENT

Before much work was done on heater development, upper limits of temperature were set at approximately 1200°C for the oven and about 1225° for the arc chamber region. No difficulty was experienced in attaining the desired

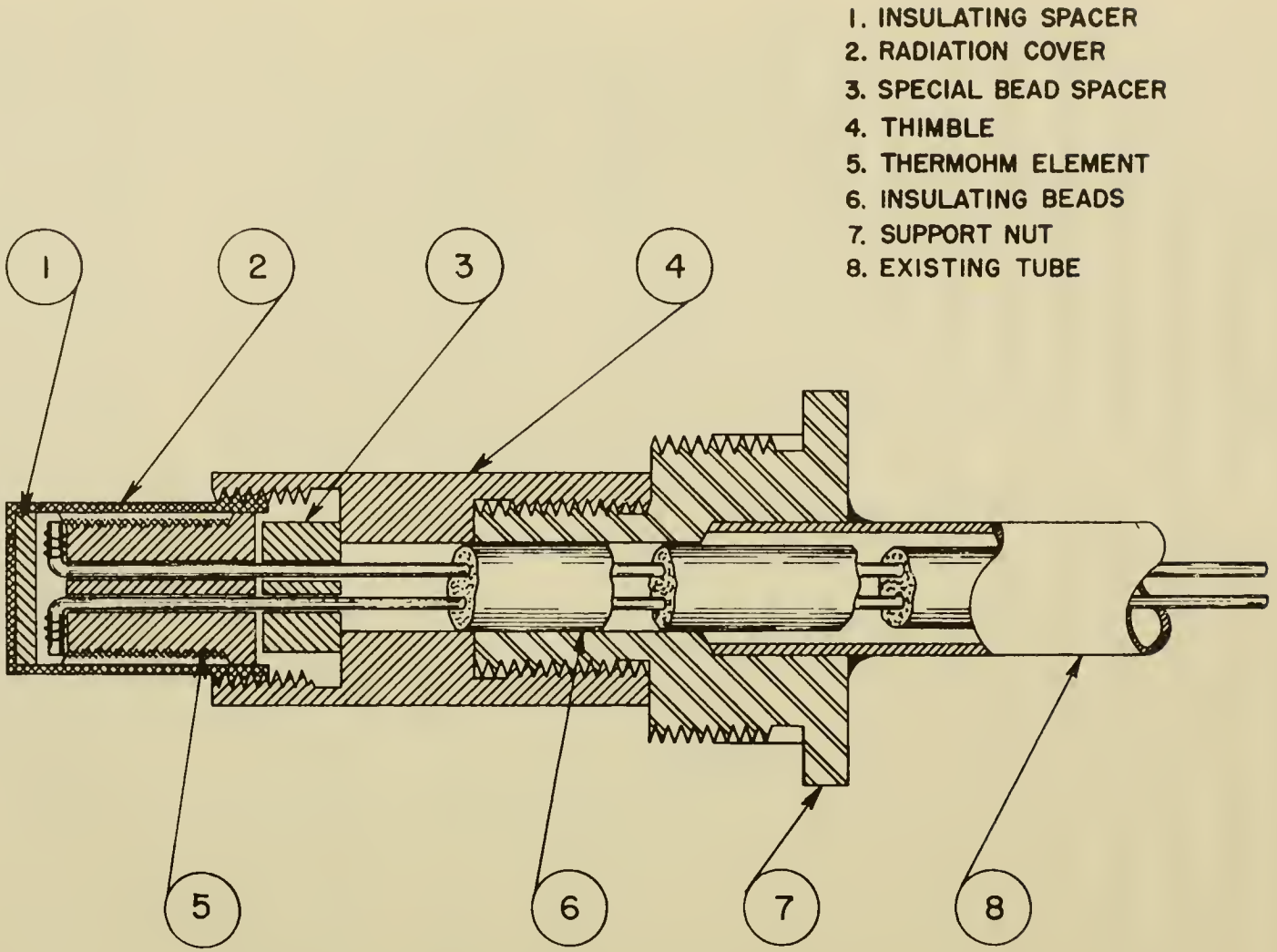

FIGURE 1. HIGH TEMPERATURE "M" SPECIAL THERMOHM ASSEMBLY

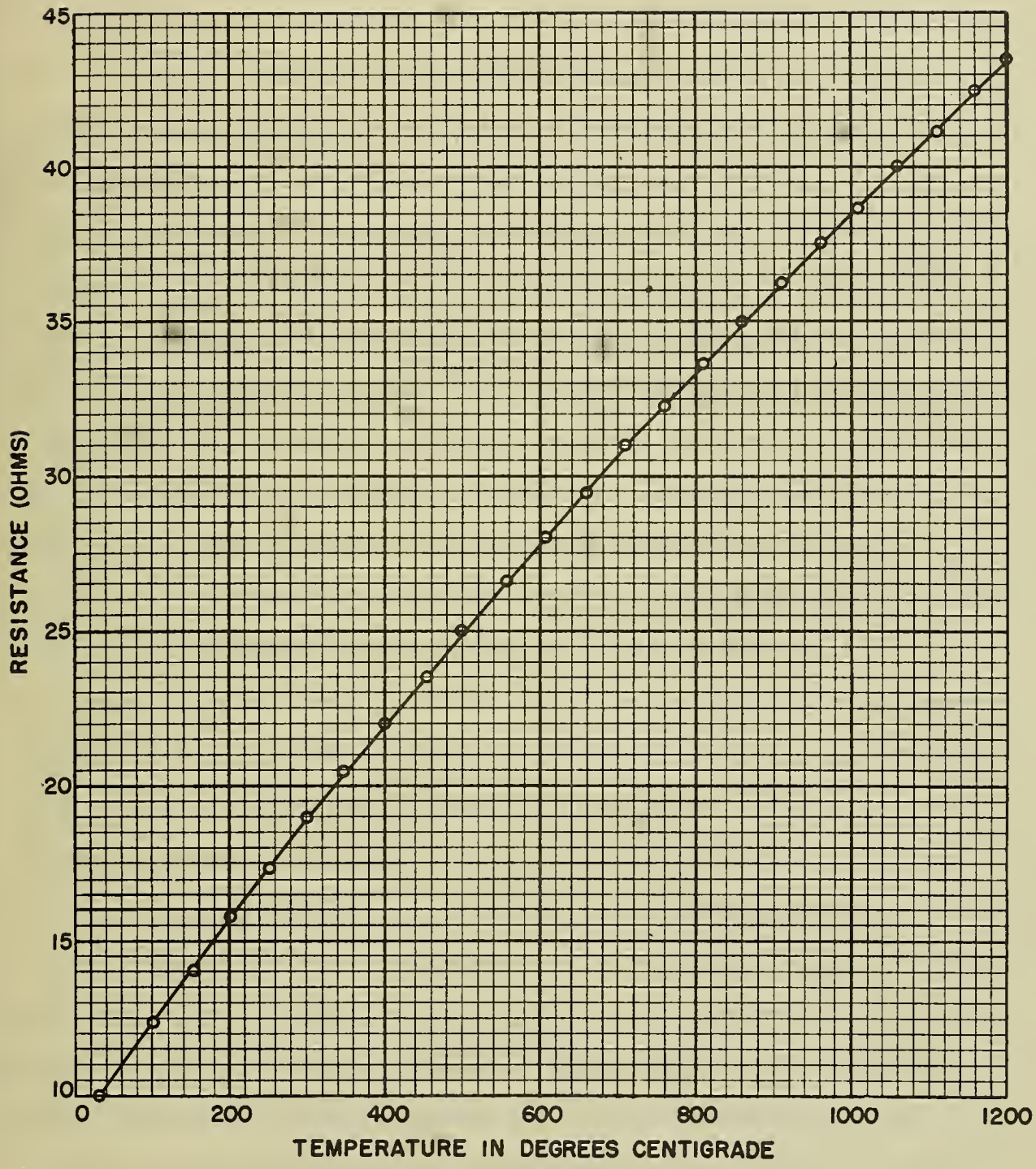

FIGURE 2. PERFORMANCE CURVE OF HIGH TEMPERATURE THERMOHM

oven temperature with the original design, but considerable work was necessary to attain the desired temperature in the arc chamber.

The first difficulty encountered with the unit as originally redesigned resulted from excessive heat loss by radiation from the arc chamber system. The arc chamber heaters were enclosed in a graphite system which had about 60 square inches of radiating surface, and since graphite is a relatively good heat conductor, this entire surface approached the temperature of the charge vapor manifold. Under this condition maximum temperatures of only 700 to 800°C were attained with 1800 watts total heater power. This situation was partially remedied after considerable development work by separating and heat shielding the arc chamber system from the remainder of the source front. This change reduced the radiating area by a factor of about 5.

At the same time, difficulty was experienced with heater life. The original arc region heaters were rated-not at 900 watts each, but at 240 watts each-and these failed rapidly. The size and length of the molybdenum wire was recalculated, and a new set of heaters fabricated according to the altered specifications. These heaters also failed; but the cause of failure appeared to be the existence of a "hot spot" caused by the proximity of the oven heater, poor heat conductivity of the alundum, and overlapped heat shielding at the center of the anti-drain heater rather than to overloading. Various heater design changes failed to remedy the situation; thus, a structual change was made in the arc system. The heated region was modified to surround the heater on three sides with graphite, thereby facilitating heat transfer from the heater to the arc system. Further heat shielding was added around

the assembly; and as a result, temperatures greater than 1000°C were obtained.

Tests were initiated to determine the heater life in the redesigned structure. Heater failure occurred in about 20 hours (max.) at 1800 watts of power input and arc region temperatures of 900 to 1100°C. Examination of the heaters after failure revealed that the molybdenum elements were operating at temperatures in excess of 2000°C and were being vaporized. At the same time, the alundum cement was fusing in the region of the elements; and, due to the consequent reduction in cement volume and loss of intimate contact between the alundum and molybdenum wire, the heat transfer from the wire element to the alundum was reduced. Thus, a further increase in the wire temperature was required in order to maintain the desired temperature. These difficulties led to the abandonment of the molybdenum wire-in-ceramic type of arc region heater.

A series of tests were run on a graphite arc chamber electrically insulated by Lava A from the remainder of the ion source unit and heated by the passage of current from a filament transformer. Although the graphite temperature exceeded 1000°C, the temperature of the Lava was between 800°C and 900°C. This temperature was considered inadequate as charge condensation could occur on the Lava forming the wall of the vapor manifold. Consequently, this approach was abandoned.

Development of satisfactory heaters with the same physical dimensions as the wire-in-ceramic type was next undertaken. Graphite elements were employed; and various insulating materials and types of heater terminations were tested. Graphite elements cast on the surface of alundum cement with

welded-on stainless steel terminals were tried; however, fabrication difficulties forced abandonment of this type. A graphite element clamped between two pieces of "steatite" was tried, but the steatite melted. Finally, graphite elements supported in Lava A insulators were tested, and after some development, were found to be satisfactory.

Results obtained on the molybdenum-in-ceramic type arc region heaters indicated that the oven heaters would be more satisfactory if constructed using graphite elements and Lava A insulators. A set of oven heaters of this type was fabricated, tested and found to be satisfactory. Figure 3 shows an oven and an arc region heater both of which were fabricated from graphite.

The tests of the thermal characteristics of the ion source were made in Tank II XBX using a nitrogen supported arc. The oven heaters were supplied with a maximum of 1800 watts and a steady power of 1500 watts, and the entire assembly was surrounded by four layers of stainless steel heat shielding. The arc chamber heaters were supplied with 1800 watts from a special 4KVA insulating transformer; and two layers of graphite, two of tantalum, and four of stainless steel were used as heat shielding around the arc system. With this arrangement, vapor manifold and arc chamber temperatures in excess of 1500°C were obtained with corresponding oven temperatures from 1000°C to 1200°C. Figure 4 illustrates the arc chamber and heater assembly without heat shielding. Higher oven temperatures were attainable by adding more heat shielding; but rapid deterioration of the stainless steel charge bottle occurred at temperatures in excess of 1200°C.

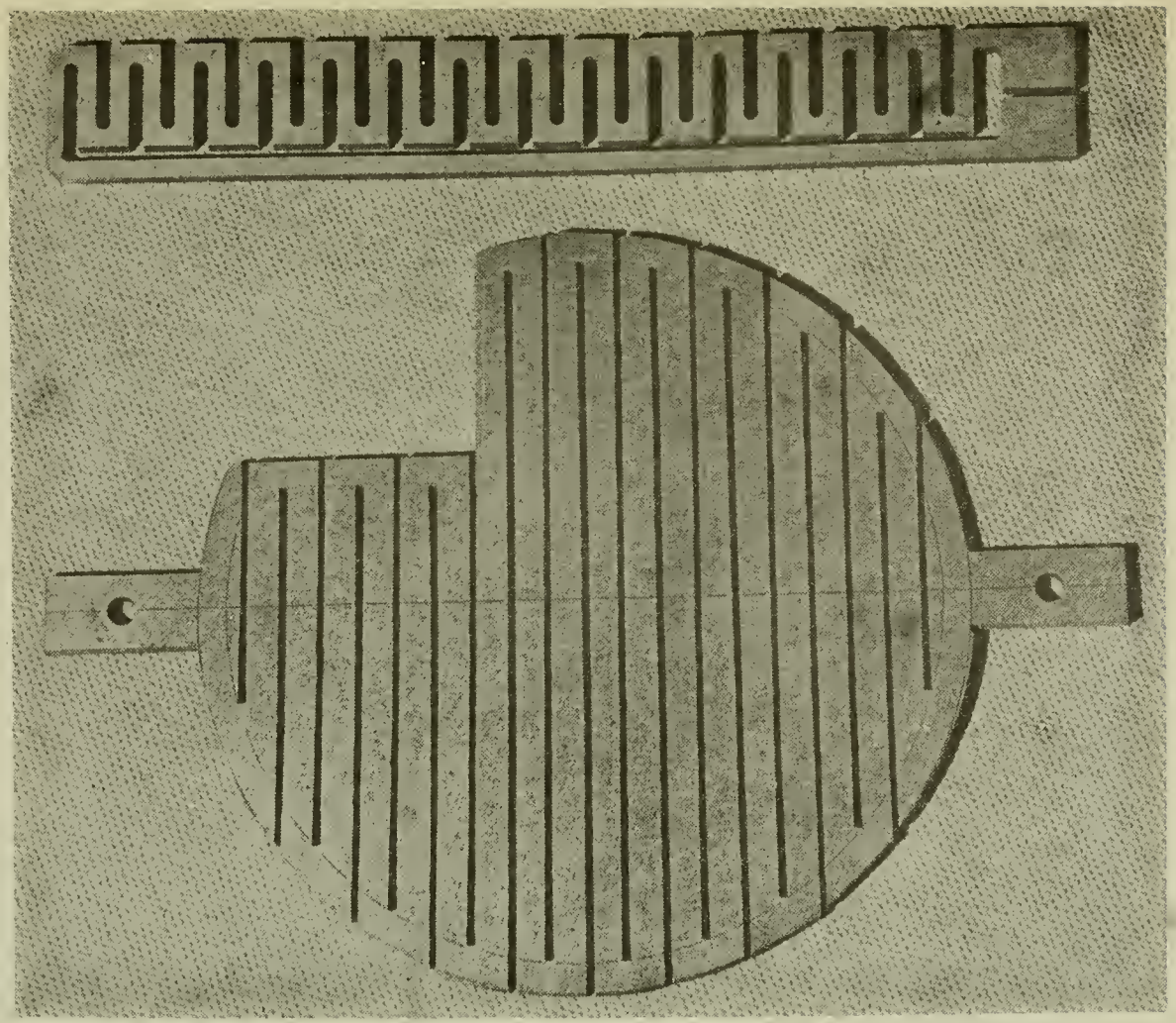

FIGURE 3. GRAPHITE OVEN AND ARC HEATERS

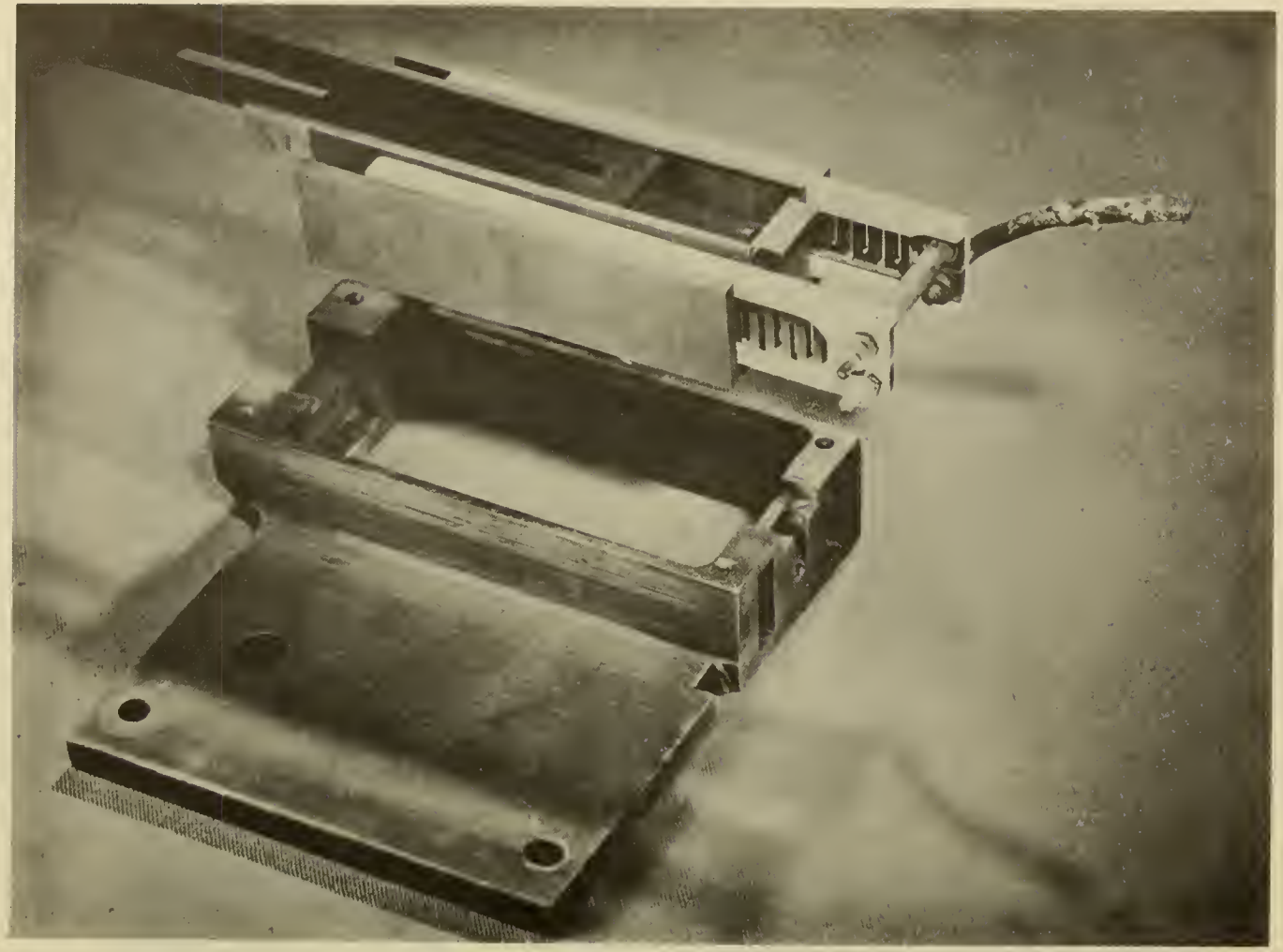

FIGURE 4. ARC CHAMBER AND HEATER ASSEMBLY

A second series of production tests was run using cerous chloride ($CeCl_3$) charge material, and difficulties encountered on these runs were:

1. Failure of arc chamber heater lead.
2. "Hunting" of the micromax and consequent oven temperature control difficulties.
3. Charge blow-out during initial pump-down.
4. Short charge bottle life.

The first of these difficulties was caused by the motor action of the heater lead in the magnetic field when currents from 30 to 36 amperes (1250 to 1800 watts) were supplied to the heaters. This was partially overcome by placing lock nuts in all possible locations and by using heavy copper leads hard-soldered to the stainless steel machine screws with a heavy fillet.

Micromax hunting was overcome by placing an extra terminal strip in the source unit terminal box for use of the thermohm leads only thereby removing them from the vicinity of the heater leads. This change reduced markedly the pickup in the thermohm circuit and consequent hunting of the micromax.

Charge "blow-out" during pumpdown was severe because of the unrestricted path between the bottle and arc exit slit. Baffles were inserted in the bottle throats and manifold to reduce this effect; but it was found advisable to reduce the initial pump-down rate with a restricted roughing line.

Stainless charge bottles were found to have a limited life (two runs) and several bottles were fabricated from graphite which proved to be satis-

factory. Figure 5 shows a graphite charge bottle and oven heater assembly.

CONCLUSIONS

A source using graphite heating elements supported on Lava A insulators is now available for use with charge materials whose operating temperatures are in the range from 650°C to 1000°C, and a thermohm suitable for use in this temperature range has been developed.

ACKNOWLEDGMENTS

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C. E. Normand and J. R. Patton cooperated in the preparation of this report.

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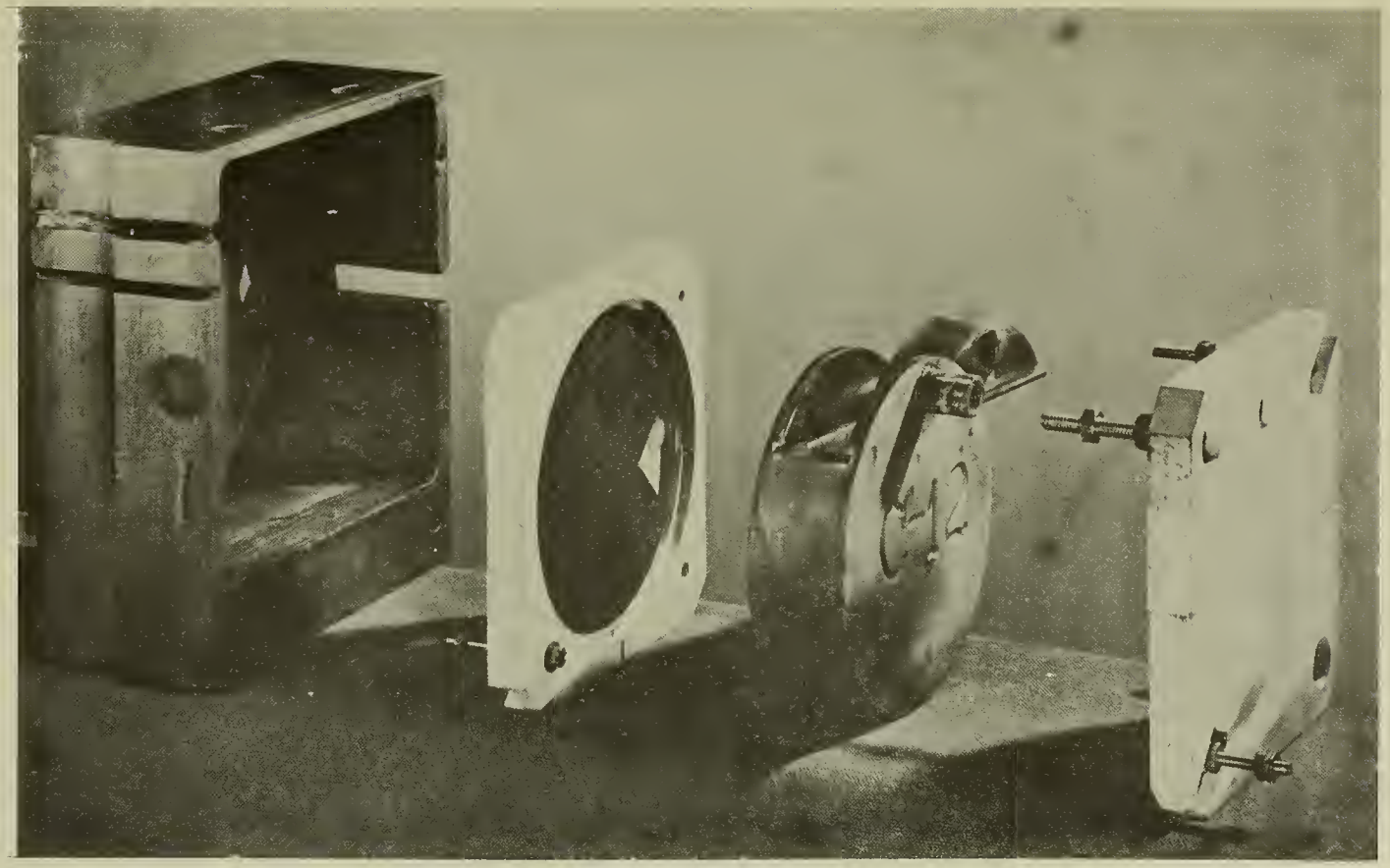

FIGURE 5. GRAPHITE CHARGE BOTTLE AND OVEN HEATER ASSEMBLY